IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re patent application of

Mark J. Sabourin

Serial No. 08/907,687

Examiner: M. Alvo

Filing Date: August 8, 1997

Group Art Unit: 1731

For: **Method of Pretreating Lignocellulose Fiber-Containing Material for the Pulp Making Process**

Commissioner for Patents
Washington, DC 20231

Sir:

DECLARATION OF MARC SABOURIN

I, the undersigned Marc Sabourin, am the inventor named in the subject patent application. I am presently the manager of the R&D laboratory of Andritz Inc, in Springfield, Ohio, and have held that position since September 1999. Andritz Inc. is the second largest worldwide supplier of thermo-mechanical pulping (TMP) refiners

1. I have been employed in research and development relating to the conversion of wood to pulp, for over fifteen years. During that time, I have been the sole or co-author of over thirty technical papers that were presented and published in internationally distributed journals, including Tappi Journal, Journal of Pulp & Paper Science, and Pulp & Paper Canada.

2. I have authored several papers reporting results of the development of the subject invention. These include (1) "Optimizing Thermomechanical Pulping of Southern Pine Species Using a Compression Pretreatment", TAPPSA Ninth Biennial International Conference (1998), (2) "Evaluation of a Compressive Pretreatment Process on TMP Properties and Energy Requirements", Pulp and Paper Canada

(2000), and (3) "Mill Scale Results on TMP Pulping of Southern Pine With Pressurized Chip Pretreatment", 87th annual Pulp and Paper Association of Canada (2001).

3. The claims of the subject application as presently amended are directed only to a method for producing thermo-mechanical pulp (TMP) from wood chips. In this regard, each of the independent claims requires three distinct operations: (1) pretreatment by conditioning and compressing the chip feed material at particular environmental conditions, (2) preheating the pretreated chip material, and (3) refining the preheated chip material. These three distinct operations and examples of associated conditions are disclosed in the specification on page 3, line 25 (pretreatment at 15-25 psi in conditioning component 3 and compressing component 6 of Fig. 1); page 12, line 5 (preheating at a temperature above Tg in preheat system 15 of Fig. 1); and page 12, line 5 (refiner 10 of Fig. 1).

4. Thus, "pretreatment" as described and claimed by applicant, is very different from "preheating". These are performed in different chambers, at different environmental conditions. The conditioning at elevated temperature and pressure and compression at elevated temperature and pressure produce a synergistic effect such that the fibers, although destructured, remain pliable, i.e., they do not become so friable as to crumble when rubbed between the thumb and forefinger. In this context, "destructured" means high levels of axial fiber separation, and "without significant breakage across grain boundaries" means most fibers retain their length in the longitudinal direction. This is illustrated in the figures of the application.

5. The improvement attributable to the pretreatment environment with saturated steam at elevated pressure (superatmospheric steam) of Example 1 of the application relative to Comparative Example 1, is set forth starting on page 20, line 24 of the specification. The significance is also demonstrated by the data and conclusions appearing in Exhibit 5 of the Preliminary Amendment filed January 10, 2001, and the discussion in the Remarks portion of that preliminary Amendment.

6. In the Preliminary Amendment filed January 10, 2001, applicant emphasized in the remarks, and gave an example in the paper submitted as Exhibit 5, that practitioners in the relevant field routinely use "psi" when referring to "psig". In my experience authoring numerous technical papers and engaging in technical discussions with scientists and customers, when referring to operating a plant, "psi" does not default to "psia", rather "psi" defaults to "psig" and in the exceptional circumstance when absolute pressure is of significance, the unit "psia" would be employed. I have attached as an example, a copy of the paper entitled "The Effects of Running Pressures on TMP Properties in a Two-Stage Process" from the Journal of Pulp & Paper Science, Volume 11, No. 1, (January 1985). The authors are from the Norwegian Pulp & Paper Institute, and as is typically in Europe, the pressures as specified on page J30 (80 to 180kPa corresponding to 116-131 degree C) do not carry a further indication of gage or absolute. This is because readers would clearly understand that a pressure specified in a plant-operating context, defaults to gage pressure. Moreover, for the particular values as specified in the paper, 80kPa = 11.6 psi = 26.3 psia, which corresponds to 116°C in a steam table reference. Similarly, 180kPa must equal 26.1 psig, which converts to 40.8 psia and this is the pressure corresponding to 131°C as taken from such steam table. The pressures shown in the graphs in Figures 5, 6, 7, 10, and 11, 12 and 13 would similarly be understood as referring to gage pressure. I note that the correct unit expression for pressure in the metric system is a pascal, which is abbreviated as Pa; therefore the "a" does not refer to absolute in this unit.

7. I also refer to page 17, line 14 through page 21, line 6 of my patent specification. It is clear that the purpose of the discussion and associated Table A, is to show that Example 1 (pretreatment in a 22 psi and 128°C saturated steam environment) produces noteworthy improvement relative to Comparative Example 1 (pretreatment in an atmospheric saturated steam environment). The pressure of 22 psi can only mean 22 psig, because only 22 psig corresponds to the specified saturated temperature of 128°C in Table A. The Examples in the specification provide the only disclosure of a specific pressure and associated specific temperature. Thus, if 22 psi corresponds to

128°C, in the Example, and the saturation temperature at 22 psig is 128°C, then one of ordinary skill would realize that throughout the specification, "psi" means "psig".

8. Furthermore, the Table in the Preliminary Amendment filed January 10, 2001 contains several data points from a technical paper (number (3) in the second paragraph above) in which the present inventor was the principle author, showing that when "psi" is used in this field of technology, gauge pressure is intended.

9. In my professional opinion, the description enables any person skilled in the relevant field, to make and use the claimed invention. Such person would learn that pretreatment <u>before preheating and refining</u>, comprises steaming at saturated steam conditions above atmospheric pressure, followed by high compression in a saturated steam environment above atmospheric pressure. He would learn that for the process condition that is most easily controlled, i.e., pressure, the magnitude should be at least 10 psi but not high enough to produce significant darkening of the lignin, and preferably in the range of 15-25 psi.

10. The glass transition temperature (transition in viscosity) of lignin, is not directly correlated with thermal darkening reactions. Although certain molecules in the lignin do darken as the temperature of the lignin rises, researches have not established any link between viscosity and thermal darkening. According to the seminal paper, "On the Characterization of Pressurized Refiner Mechanical Pulps", by D. Atack of the Pulp and Paper Research Institute of Canada, presented at the International Symposium on Paper Pulp Characterization, Ronneby, Sweden, May 24 - 26, 1971, "The glass transition temperature, often referred to as the softening temperature, is a characteristic temperature at which an amorphous polymer such as lignin undergoes a major reversible structural transition from an almost elastic glassy state to an almost elastic rubbery one." The author states that the glass transition temperature is dependent on the bar crossing frequency in the refiner, and that "it may be estimated that under refining conditions lignin in moist spruce chips softens somewhere in the range 120 – 135 degree C."

11. Thermal darkening is a function of temperature, and time at temperature. Thermal darkening reactions can occur starting at about 100°C, which is below the typical glass transition temperature. Moreover, the glass transition temperature itself for a given material can vary depending on, as a one variable, the stress of the material during compression and the beat frequency experienced by the material during mechanical refining. Stressing the material in this way evidently raises the glass transition temperature.

12. Thus, in my opinion, whether the material temperature during pretreatment according to my invention rises into the range of 120-135°C is not the sole, critical factor in discoloration. Tg in this context is merely an indication that the material is at a temperature where care must be taken regarding the time exposure to avoid excessive thermal darkening. It is not a rapid discoloration effect that occurs once the lower end of the Tg range is reached.

13. Nevertheless, the invention encompasses a temperature of the material into the range of Tg because under compression we want to separate the fibers at the fiber walls. Significant lignin is present in the fiber walls. Thus, the lignin should be soft enough that separation will occur at the lignin, but the combination of time and temperature at these elevated temperature conditions should fall within a window of effectiveness. In my invention, the purpose of operating at superatmospheric pressure is to produce a temperature in the material that will significantly soften the fiber such that fiber separation will occur without rupturing the fiber walls, while avoiding such high temperature that there will be an associated significant degradation in color. The claimed range of 10-25 psi specifies this most effective window with 15-25 psi being especially preferred.

14. In contrast, for chemical pulping, there is no concern about darkening reactions in the lignin during upstream processing, because the chemical treatment in essence dissolves the lignin, which is no longer present in the pulp. In TMP refining,

however, the fibers are separated from each other but lignin material remains with the separated fibers and if any of the processing produces discoloration, this will be evident in the resulting pulp. This is the reason why, in my opinion, practitioners in the realm of chemical pulping would not be expected to think carefully about the effect of upstream processing on the lignin in the feed material. Similarly, practitioners in the field of TMP would not look to upstream processes associated with chemical pulping, such as disclosed in Prusas, because they would not expect to find any teaching in the chemical pulping that would assist them in improving pre-processing with attention to minimizing discoloration due to lignin darkening.

15. Furthermore, in order to maintain the quality of the resulting fiber, breakage of fibers across grain boundaries is to be avoided, notwithstanding that the chip material undergoes tremendous stress in the screw press. The elevated temperature associated with superatmospheric pressure in the specified range claimed by applicant, not only enables the fibers to separate, but this preferential separation avoids the excessive stresses across grain boundaries and therefore produces the effect recited in claim 31, i.e., "destructuring said fibers without significant breakage across grain boundaries". In the examples given in the Prusas patent, there was significant breakage across grain boundaries. In my opinion, even if a suggestion were made to one of ordinary skill in the field of TMP, to the effect that pretreatment could be accomplished in a compression screw running at the very high temperature condition suggested by Prusas, such ordinarily skilled practitioner would immediately realize the danger of discoloration and would not have reason to analyze the situation further to arrive at a window of pressure in the range of 10-25 psi. In my opinion, it would not have been obvious to recognize that the combined benefit for TMP pretreatment of separation of the fibers and avoidance of significant breakage across the grain boundaries, without significant discoloration, could be achieved simultaneously.

16. None of the prior art shows a TMP system or process, having the three steps of (1) pretreatment of wood chips, (2) preheating of wood chips, and (3) refining of wood chips, wherein the pretreatment is performed at superatmospheric pressure, let

alone at pressures in the range of 10-25 psi. My invention concerns the pretreatment of wood material, particularly wood chips. Such material is different from pulp. According to the well-accepted definition, "pulp" is a distribution of fibers with sufficient exposed cellulosic material for interfiber bonding.

17. Pulp for papermaking can be produced from wood chips entirely by one or more series of mechanical refiners (e.g., to produce TMP pulp), or by digestion or cooking processes (chemical pulp). Commonly, chemical pulp is further processed by mechanical refining at atmospheric pressure, as a finishing step, before bleaching. This is exemplified by U.S. Patent No. 5,244,542 "Pulp Treatment methods", (Minton) where the process for producing pulp from chips by cooking (e.g, discussed at column 1, line 68 to column 2, line 4) is identified as a background step to the claimed processing of such <u>pulp</u> to produce the desired "kinks" in the <u>pulp</u>. In my professional opinion, a screw press operating even at high compression ratio of 8:1, could not produce permanent twists and kinks in the fibers of wood <u>chips</u>.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and that the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: April 10, 2002

Name: [signature]
Marc J. Sabourin

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The Effects of Running Pressures on TMP Properties in a Two-Stage Process

by J.W. BRILL and S. HAUAN

In order to make the best use of the steam generated in the TMP process, many mills today want to increase the refining pressure. Results from trial runs in different two-stage, single-disc TMP plants show that an increased difference between the housing and the feeder zone pressures apparently promotes an increased disc clearance. The cause of this is discussed in the text. The effect on the pulp quality is small. At equal freeness levels the strength properties seem to increase slightly at a cost of a decreasing scattering coefficient. The long fibre content is also affected to some degree: for low preheating pressures it decreases, for higher preheating pressures it increases.

INTRODUCTION

Yesterday's complaints against the high energy consumption of the TMP process seem to have diminished. The main cause of this is a better utilization of the generated steam. Most modern TMP-mills run with pressurized refiners, because the increased pressure gives a higher value to the steam. An interesting question is, therefore, how does this influence the refining process and ultimately the pulp quality?

In Norway pressurized refiners are only found in the first stage of two-stage systems. There are three such plants. The two new ones (from 1981) are equipped with Sunds Defibrator 58" refiners and the oldest one (from 1977) with Jylhävaara 48" refiners.

The Norwegian Pulp and Paper Research Institute (PFI) has a project going where one of the aims is to investigate the effects of different running pressures on refining parameters and pulp quality. Trial runs have been made in the above-mentioned mills and more are planned. The data discussed in this paper are based mainly on a trial series made at Norske Skogindustrier's TMP-mill in Skogn. The conclusions are, however, backed up by data from other trial runs.

EXPERIMENTAL

Fig. 1 shows a rough sketch of the refiner system in Norske Skogindustrier's TMP plant. In the first stage there are 4 Sunds Defibrator RLP 58 pressurized refiners and in the second stage 3 RL 58 open discharge refiners. The disc diameter is 1450 mm. Under the trial runs only refiners numbered 1, 2 and 6 were used. Pulp samples were collected from the blow pipes of the first stage refiners and from the pulp channel under the second stage refiner.

The trials were runs using chips from Norway Spruce (sawmill chips mostly). The chips were screened through a 45x45 mm grid to remove oversize chips. In order to estimate the chip quality variations, samples were collected before and after series of trial runs. The analyses of these samples showed a variation of the fines content between 0.6 and 3.1% measured in a STFI-type chip classifier with a 3 mm hole bottom screen. This, undoubtedly, introduces an unknown degree of uncertainty in the results.

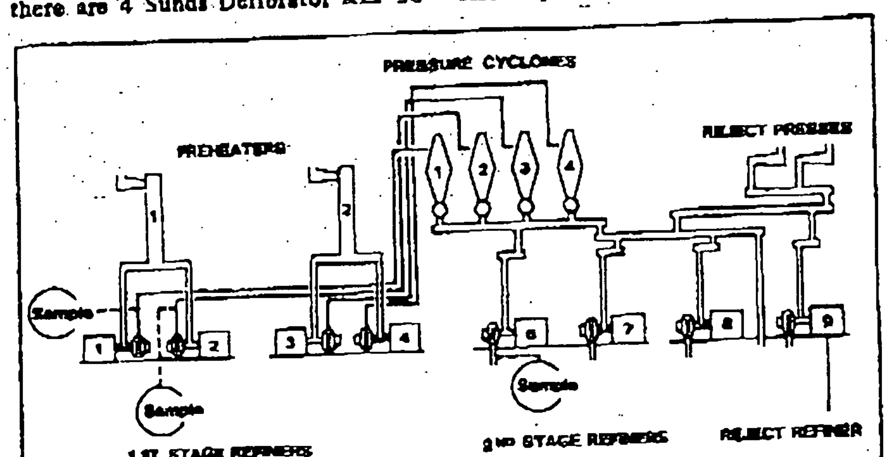

Fig. 1. The refiner section of the Norske Skogindustrier's TMP plant.

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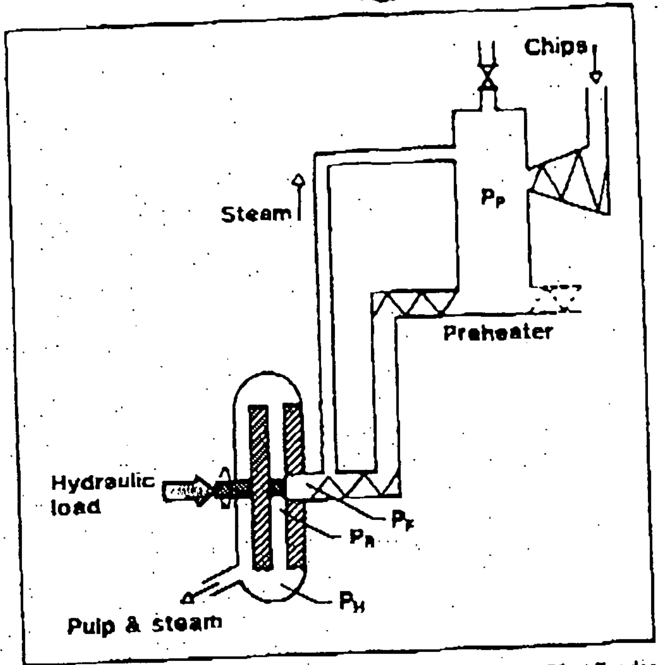


Fig. 2. The pressures in the first stage refining system. Pf = Feeder zone pressure; P_H = Housing pressure; Pp = Preheater pressure; P_r = Refining zone pressure.

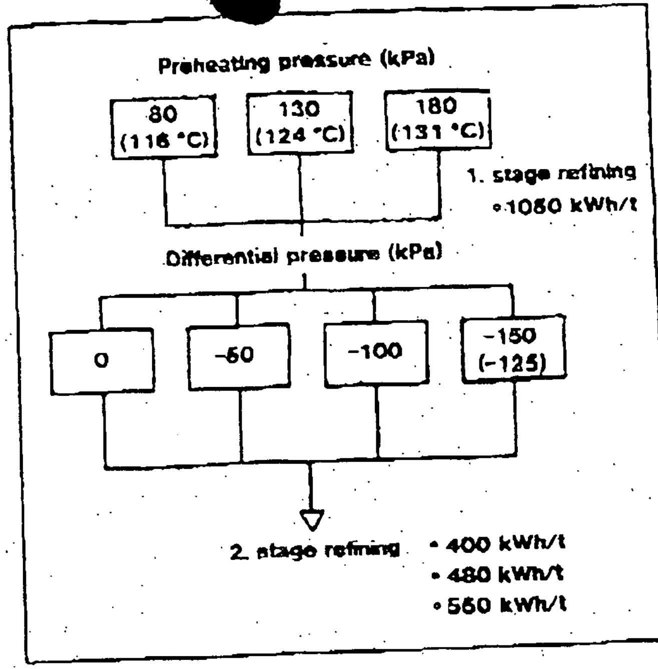


Fig. 4. The trial run scheme.

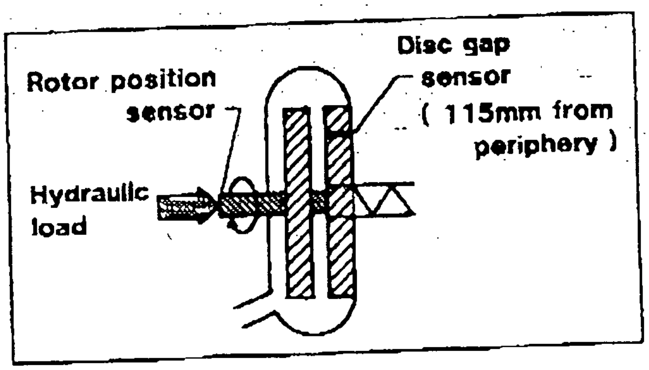


Fig. 3. The disc clearance sensors.

The first stage refining system is schematically shown in Fig. 2 The different pressures are here defined. The "disc gap" sensor and the rotor position sensor are shown in Fig. 3. The differential pressure is defined in this paper as the preheater pressure minus the housing pressure. For the trial runs described, the differential pressure is always negative. (We have to assume that the preheater pressure is of approximately the same magnitude as the feeder zone pressure, because we did not have means to measure the latter.)

The trial-scheme is showed in Fig. 4. While keeping the preheater pressure at constant levels (80 to 180 kPa corresponding to 116-131°C), the housing and in consequence the differential pressure was altered (between 0 and -150 kPa). The specific energy was held at a constant level in the first stage, while three different levels were run in the second stage. The production rate was at all times constant. The discharge consistencies varied between 35 and 50% for the first stage refiners and between 21 and 25% for the second stage refiner. The disc segments were considered to be in normal condition.

All the pulp analyses were made according to the SCAN-test norms.

RESULTS AND DISCUSSION

Refining Parameters

What happens in the refiner when the running pressures are altered? The trials have shown that at an increasing differential pressure, the hydraulic load is reduced. As shown in Fig. 5 there is a linear relationship between these parameters. This relationship seems to be independent of the pressure level of the preheater. The hydraulic load was dropping 1 kN for each kPa increased differential pressure. The cause of this reduction is obvious. If the feeder zone pressure is kept constant, an increased housing pressure will press the rotor disc against the stator disc, thus reducing the need for hydraulic load in order to keep the energy consumption constant. The disc area is approximately 1.6 m^2. Assuming that the feeder zone and the preheater pressures are of the same magnitude (the feeder zone pressure will of course be somewhat higher), this means for each kPa increased differential pressure, a force of 1.6 kN is applied on the rotor disc. Subtracting the hydraulic load drop of 1.0 kN from 1.6 kN, the average pressure rise between the discs can be calculated to 38 kPa for each 100 kPa rise in the differential pressure.

Increasing the differential pressure led to an apparent increase in the disc clearance at a constant energy consumption. The measured values for the "disc gap" and the rotor position are shown in Figs. 6 and 7. The development of the rotor position is in agreement with the results Sundholm and Mannström have reported from their PRMP-trials [1], although these

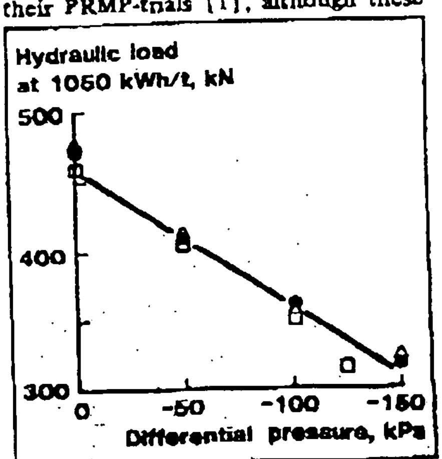


Fig. 5. Hydraulic load vs increased differential pressure at constant energy. • 80 kPa preheating pressure; ▲ 130 kPa preheating pressure; □ 180 kPa preheating pressure.

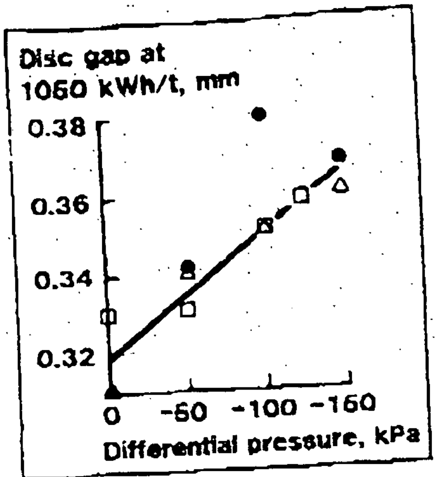


Fig. 6. Disc clearance measured by the "disc gap" sensors vs the differential pressure at constant energy.
• 80 kPa preheating pressure; △ 130 kPa preheating pressure; □ 180 kPa preheating pressure.

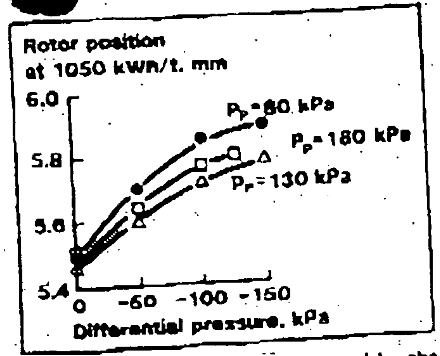


Fig. 7. The disc clearance measured by the rotor position sensor vs the differential pressure at constant energy.
• 80 kPa preheating pressure; △ 130 kPa preheating pressure; □ 180 kPa preheating pressure.

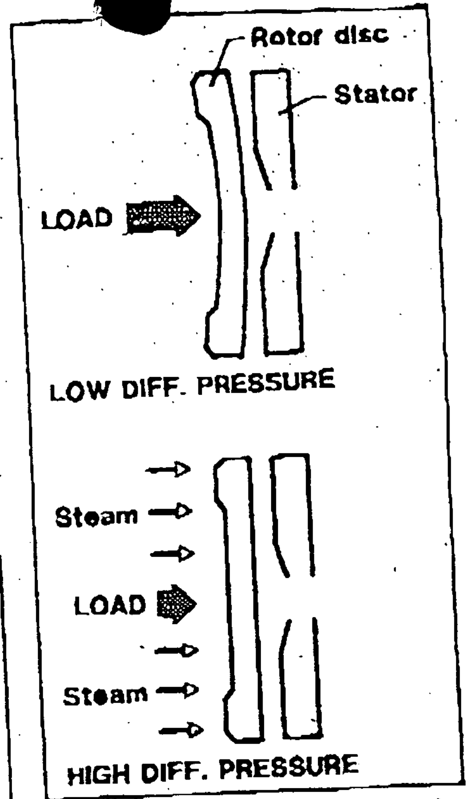


Fig. 8. The supposed influence of the differential pressure on disc parallellism.

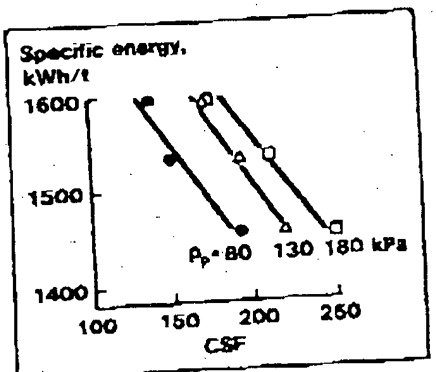


Fig. 9. Specific energy consumption vs CSF at different preheater pressures.
• 80 kPa preheating pressure; △ 130 kPa preheating pressure; □ 180 kPa preheating pressure.

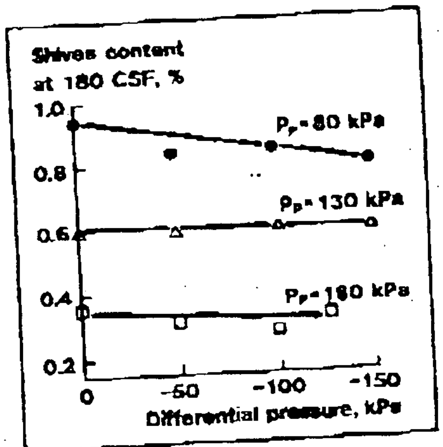


Fig. 10. Shives content at 180 CSF vs the differential pressure at different preheater pressures.
• 80 kPa preheating pressure; △ 130 kPa preheating pressure; □ 180 kPa preheating pressure.

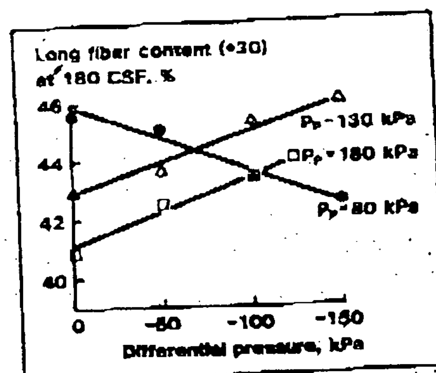


Fig. 11. Long fibre content at 180 CSF vs the differential pressure at different preheater pressures.
• 80 kPa preheating pressure; △ 130 kPa preheating pressure; □ 180 kPa preheating pressure.

trials were made on a double disc refiner.

Why should an increased pressure difference lead to increased disc clearance? A possible answer to this is higher disc parallellism. Let's look into the refiner at zero housing pressure: Now, the total load on the rotor disc is applied through the rotor shaft. Since the wall-to-wall distance in the central parts between the discs, i.e. in the breaker bar zone, is much greater than along the bar perimeter, it is obvious that the rotor disc will bend. This is schematically illustrated in Fig. 8:

An increased differential pressure will, as mentioned above, lead to reduced load on the shaft ("hydraulic load") and a more evenly spread load on the total disc area. This obviously will straighten the rotor disc, which again leads to a higher degree of disc parallellism. This could explain the increased disc clearance measured by the rotor position sensor. The disc clearance measured by a "disc gap" sensor would be very dependent on the sensor's position. If it is situated very close to the disc perimeter, a decreasing clearance would be expected, more towards the disc centre an increasing clearance would be expected.

The energy consumption to a given freeness seemed to be more or less unaffected by an increasing differential pressure. Possibly there is a slight increase which probably is compensated by the higher value of the produced steam. The pre-heater pressure affected the energy consumption as expected. This is shown in Fig. 9.

PULP QUALITY

If it is true that increased differential pressure leads to a higher degree of disc parallellism, one would expect a better and gentler treatment of the fibre material, i.e. more fibrillation and less fibre shortening. The results from the pulp analysis partially confirm this, even if the changes are relatively small. The properties of the second stage pulp are in the subsequent sectors compared at a given freeness.

While the shives content was slightly reduced at increased differential pressure (Fig. 10), the long fibre content seemed to increase with increasing differential pressure for 130 and 180 kPa preheater pressures and decrease for 80 kPa preheater pressure (Fig. 11). This was the case for both +30 and +14 fractions. The 80 kPa curve can probably be explained by increased fibre cutting due to unstable refining caused by increased back-flow steam volume.

The tensile strength as shown in Fig. 12 seemed to increase slightly at higher differential pressures. (It has been seen also in other industrial trials

that low preheating pressures can give stronger pulps than higher pressures. This will be studied further in future trials.) The tear strength as shown in Fig. 13 followed the tensile strength to a maximum at about 100 kPa, but differentiated the 80 and 130 kPa pulps due to a different long fibre content (see Fig. 11). The light scattering decreased slightly at high differential pressures and more with decreasing preheater pressure. Earlier trials, however, have shown increased light scattering at higher differential pressures so no clear conclusions can be drawn. The brightness was not significantly affected by the running pressures.

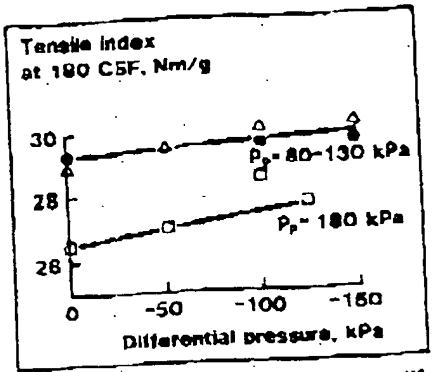


Fig. 12. Tensile index at 180 CSF vs the differential pressure at different preheater pressures.
• 80 kPa preheating pressure; △ 130 kPa preheating pressure; ○ 180 kPa preheating pressure.

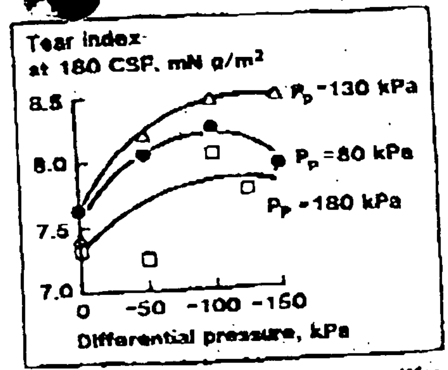
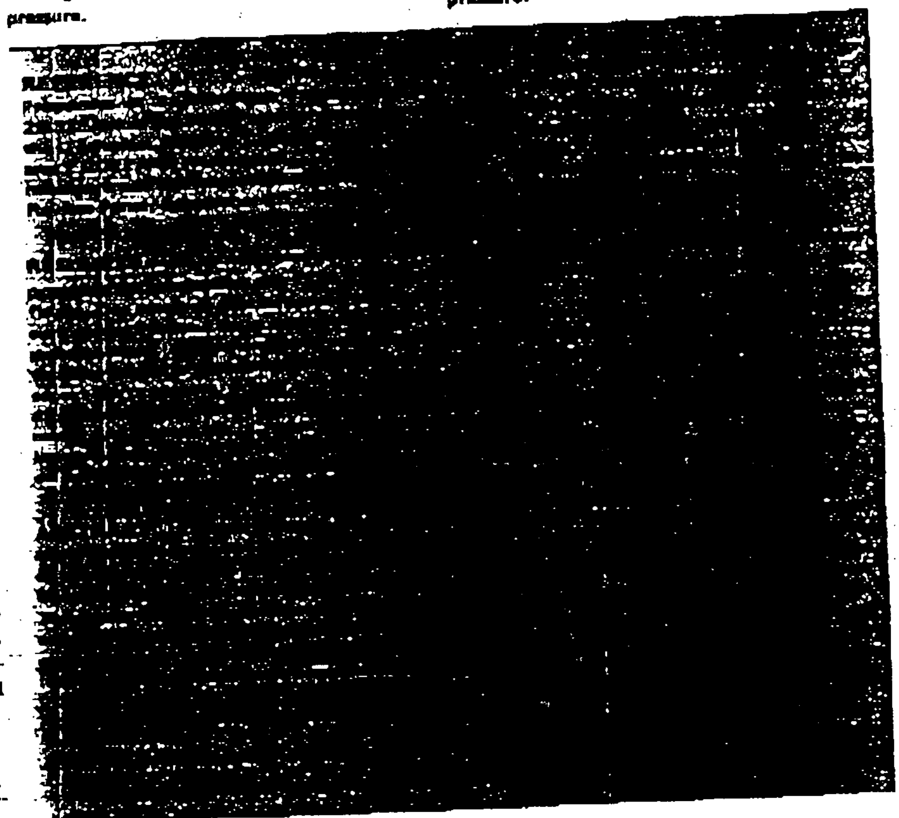


Fig. 13. Tear index at 180 CSF vs the differential pressure at different preheater pressures.
• 80 kPa preheating pressure; △ 130 kPa preheating pressure; ○ 180 kPa preheating pressure.



CONCLUDING REMARKS

Within the examined pressure range, the trials have shown that increasing difference between the housing pressure and the feeder zone pressure possibly promotes a better disc parallellism. The pulp quality is slightly affected as follows. At equal freeness the tensile and tear is somewhat higher, however at the expense of a decreased light scattering. The shives content is reduced and the long fibre content is increased or decreased, dependent on the preheater pressure level.

These conclusions are mainly in accordance with experiences made in other trial runs on Sund Defibrator and Jylhävaara plants. However, more trials will be run after the writing of this paper.

ACKNOWLEDGEMENT

This work is mainly based on mill trials at Norske Skogindustrier A/S. The authors wish to thank the company and the company staff for all goodwill and cooperation.

REFERENCE

1. J. SUNDHOLM and B. MANNSTRÖM. *Paperi ja Puu* 64 (1) : 8-13 (1982).

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